

EVALUATION (D-SPACINGS, RELATIVE INTENSITIES) OF POWDER X-RAY DIFFRACTION PATTERNS

Equipment used

X-Ray Powder Diffractometer D-8 (AXS-BRUKER)
theta-theta-goniometer, sample changer
target: Copper, $K\alpha_1 + K\alpha_2$ $\lambda = 1.5406 \text{ \AA}$
parallel beam optics (receiving soller-slit: 0.07 mm)
Scintillation counter, standard sample holders

Samples and Data-Collection

Table / Figure

- 1) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- **phosphate**
data collection: 40kV, 40 mA, 2-40° $\theta/2\theta$, 0.01 steps, 2 seconds
- 2) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- **hydrochloride**
data collection: 40kV, 40 mA, 2-40° $\theta/2\theta$, 0.01 steps, 2 seconds
- 3) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- **tosylate**
data collection: 40kV, 40 mA, 2-40° $\theta/2\theta$, 0.01 steps, 2 seconds
- 4) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- **hydrogensulfate**
data collection: 40kV, 40 mA, 2-40° $\theta/2\theta$, 0.01 steps, 2 seconds
- 5) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- **mesylate**
data collection: 40kV, 40 mA, 2-40° $\theta/2\theta$, 0.01 steps, 2 seconds
- 6) 7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-3-vinyl-3-cephem-4-carboxylic acid- **sulfate**
data collection: 40kV, 40 mA, 2-40° $\theta/2\theta$, 0.01 steps, 2 seconds

external d-spacing standards:

- 1) NIST SRM 640A (Silicon Powder)
 - 2) NIST SRM 675 (synth. Fluorophlogopite)
- data collection: 40kV, 40mA, 2 - 50° $\theta/2\theta$, 0.01 steps, 2 seconds

Software

DIFFRAC-Plus and TOPAS (AXS-BRUKER)

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**External d-spacing and relative intensity evaluation of the powder diffractometer
(D8 – AXS-BRUKER) with NIST standards:**

SRM 640A:	28.443° _{expected}	28.446° _{measured}
	47.304° _{expected}	47.308° _{measured}
	Rel.Intensity 100 _{expected}	Rel. Intensity 100 _{measured}
	Rel.Intensity 55 _{expected}	Rel. Intensity 55 _{measured}
SRM 675:	8.853° _{expected}	8.849° _{measured}
	17.759° _{expected}	17.754° _{measured}
	26.774° _{expected}	26.778° _{measured}
	35.962° _{expected}	35.962° _{measured}
	45.397° _{expected}	45.397° _{measured}
	Rel.Intensity 81 _{expected}	Rel. Intensity 80 _{measured}
	Rel.Intensity 5 _{expected}	Rel. Intensity 5 _{measured}
	Rel.Intensity 100 _{expected}	Rel. Intensity 100 _{measured}
	Rel.Intensity 7 _{expected}	Rel. Intensity 6 _{measured}
	Rel.Intensity 28 _{expected}	Rel. Intensity 27 _{measured}

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POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid phosphate

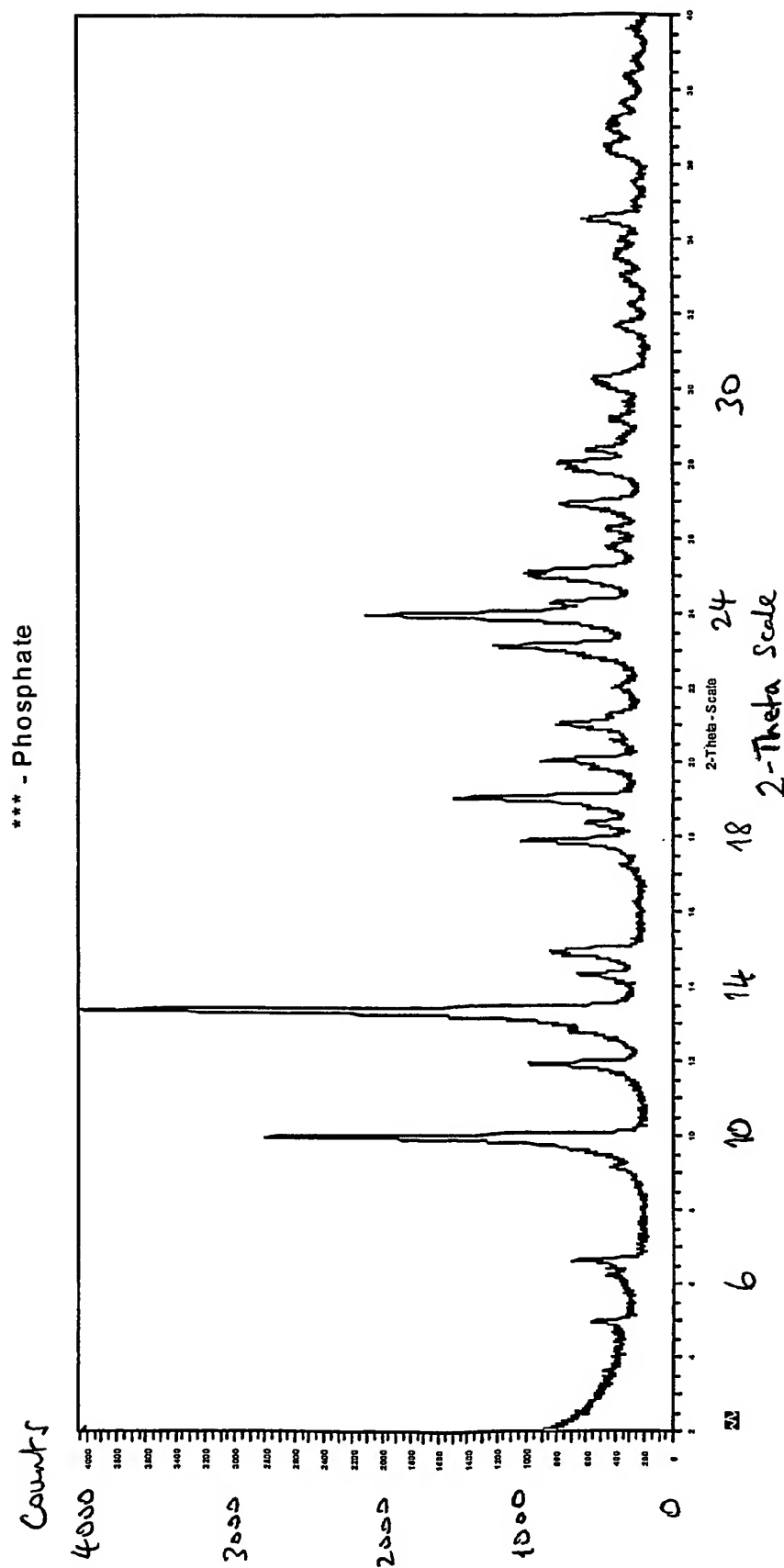
Table 1

PHOSPHATE		
d value (Angstrom)	Angle (° Cu Kα)	Intensity (Rel. Int.)
17.813	4.96	7
14.200	6.22	6
13.342	6.62	13
9.696	9.11	7
8.860	9.98	68
7.422	11.92	21
6.923	12.78	14
6.636	13.33	100
6.195	14.29	13
5.940	14.90	18
5.459	16.23	3
5.148	17.21	5
4.963	17.86	23
4.823	18.38	11
4.661	19.03	34
4.474	19.83	11
4.420	20.07	19
4.309	20.60	7
4.221	21.03	17
4.173	21.27	8
4.035	22.01	6
3.890	22.65	13
3.844	23.12	27
3.707	23.98	50
3.661	24.29	17
3.563	24.97	21
3.533	25.19	22
3.446	25.83	8
3.387	26.29	8
3.303	26.97	16
3.196	27.89	15
3.175	28.08	16
3.142	28.39	11
3.105	28.73	5
3.051	29.25	7
3.014	29.62	4
2.948	30.30	10
2.817	31.74	6
2.770	32.30	3
2.709	33.04	5
2.671	33.53	6
2.654	33.74	6
2.631	34.05	5
2.590	34.61	11
2.581	35.01	3
2.523	35.56	3
2.481	36.48	8
2.431	36.95	7
2.410	37.28	7
2.387	37.65	5
2.343	38.39	4
2.316	38.85	3
2.273	39.62	3

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POWDER PATTERN (DIFFRACTOGRAM) Figure 1

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid -phosphate



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POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid hydrochloride

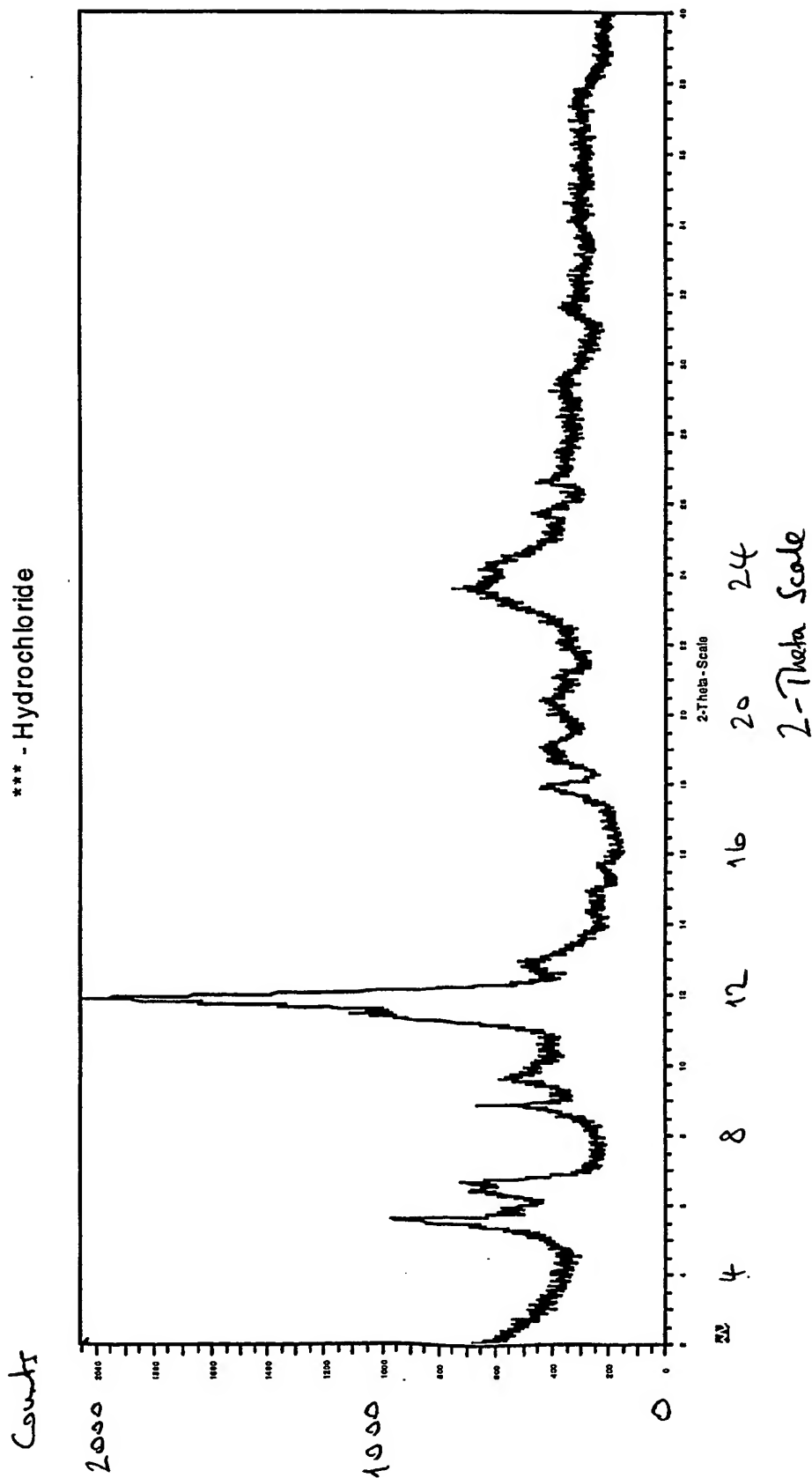
Table 2

HYDROCHLORIDE		
d value (Angstrom)	Angle (° Cu Kα)	Intensity (Rel. Int.)
15.934	5.54	36
15.176	5.82	16
13.791	6.40	23
13.281	6.65	25
9.965	8.87	24
9.171	9.64	20
7.720	11.45	48
7.460	11.85	100
6.825	12.98	17
6.129	14.44	6
5.940	14.90	6
5.661	15.64	5
4.963	17.86	14
4.744	18.69	13
4.646	19.09	13
4.406	20.14	11
4.328	20.51	13
4.197	21.15	10
4.040	21.98	8
3.760	23.65	27
3.670	24.23	22
3.454	25.77	11
3.348	26.60	11
3.238	27.52	7
3.057	29.19	7
3.017	29.59	7
2.830	31.59	7
2.752	32.51	6
2.631	34.05	6
2.404	37.38	7

POWDER PATTERN (DIFFRACTOGRAM) Figure 2

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid hydrochloride

*** - Hydrochloride



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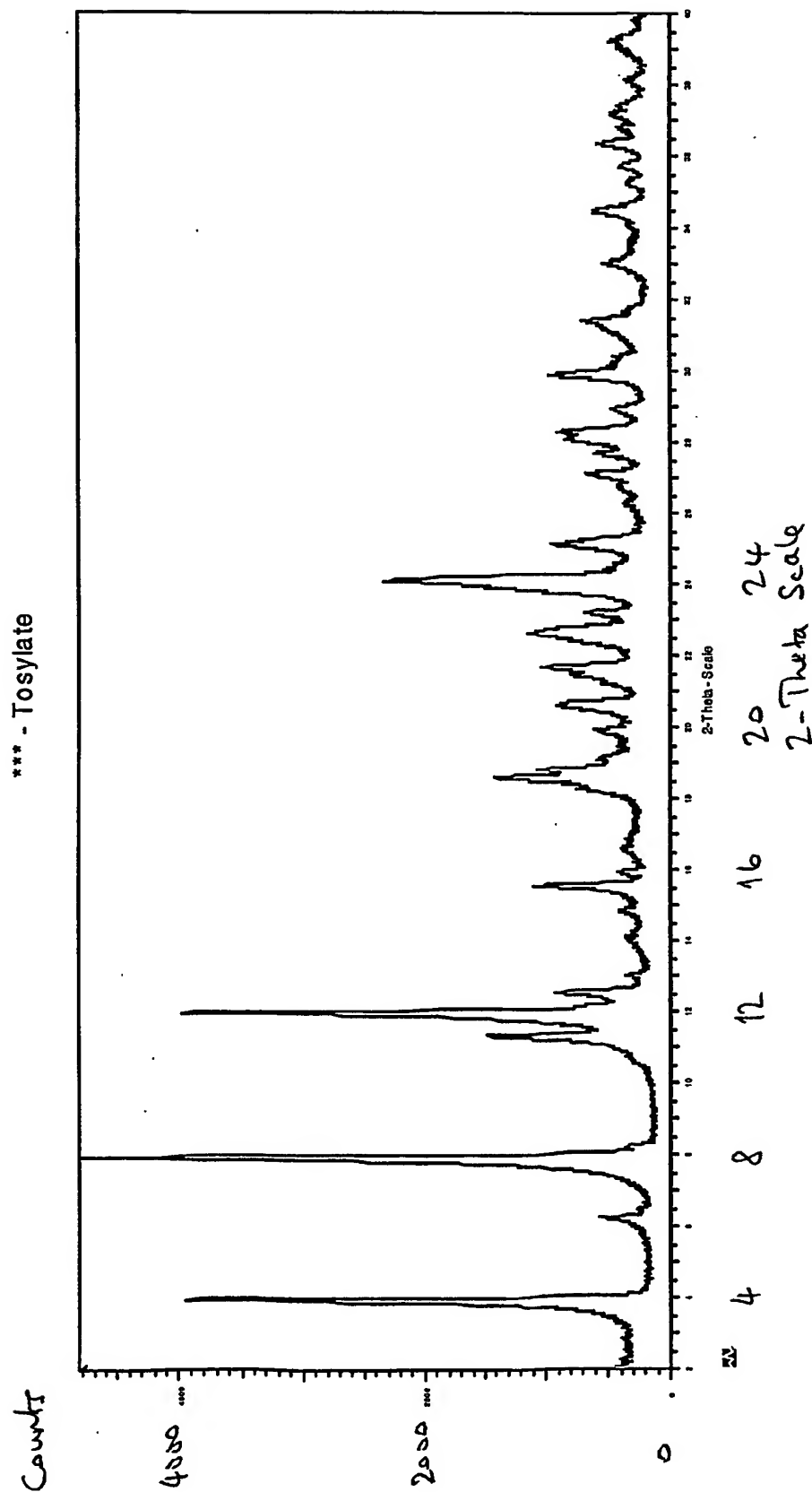
POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid tosylate

Table 3

TOSYLATE		
d value (Angstrom)	Angle (° Cu Kα)	Intensity (Rel. Int.)
22.579	3.91	80
14.130	6.26	9
13.660	6.47	3
11.165	7.91	100
10.747	8.22	5
8.322	10.62	4
7.825	11.30	28
7.403	11.96	81
7.041	12.56	16
6.777	13.05	4
6.289	14.07	4
6.977	14.81	5
5.695	15.55	19
5.553	15.95	5
5.328	16.63	4
4.847	18.29	11
4.775	18.57	26
4.714	18.81	18
4.631	19.15	8
4.460	19.89	8
4.309	20.60	14
4.143	21.43	12
4.097	21.68	17
3.926	22.63	19
3.824	23.25	10
3.717	23.92	28
3.689	24.11	45
3.642	25.12	15
3.383	26.32	3
3.329	26.76	4
3.289	27.09	9
3.213	27.74	8
3.175	28.08	13
3.145	28.36	15
3.088	28.91	6
2.989	29.86	16
2.957	30.20	6
2.936	30.42	5
2.888	31.16	8
2.844	31.43	11
2.822	31.68	4
2.714	32.97	7
2.671	33.53	3
2.628	34.08	4
2.599	34.48	9
2.542	35.28	3
2.510	35.75	5
2.471	36.33	8
2.439	36.82	5
2.417	37.16	6
2.400	37.44	5
2.381	38.09	4
2.305	39.04	5
2.293	39.26	6
2.258	39.90	2

POWDER PATTERN (DIFFRACTOGRAM) Figure 3

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid -tosylate

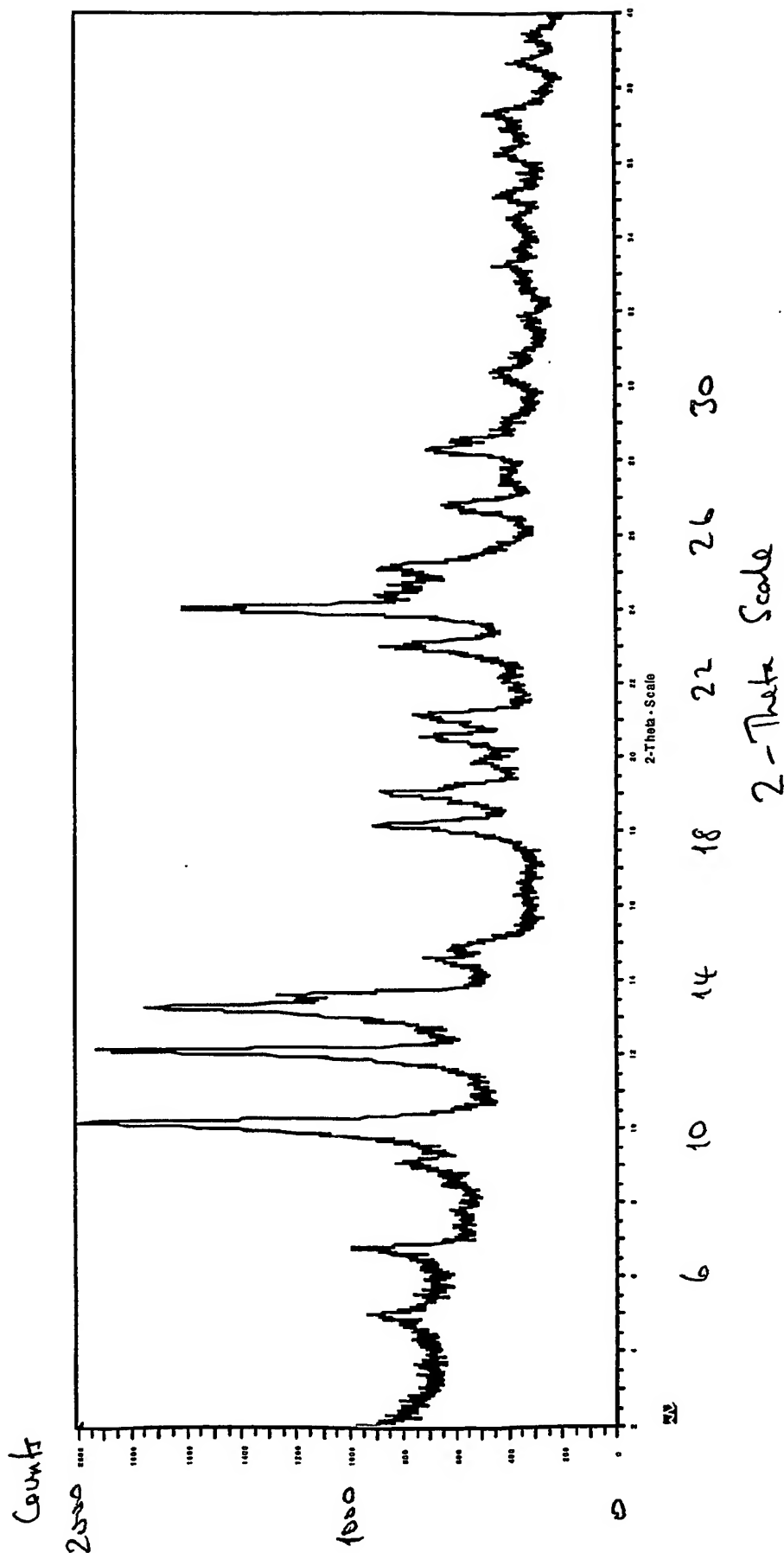


POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid hydrogensulfate

Table 4

d value (Angstrom)	Angle (° Cu Kα)	Intensity (Rel. Int.)
18.863	4.68	12
17.700	4.99	21
13.037	6.78	28
9.793	9.02	20
8.751	10.10	100
7.289	12.13	98
6.681	13.24	88
6.515	13.58	56
6.089	14.54	24
5.939	14.90	19
4.887	18.14	39
4.652	19.06	36
4.480	19.80	14
4.321	20.54	24
4.214	21.06	26
4.017	22.11	6
3.868	22.97	35
3.697	24.05	82
3.647	24.39	37
3.602	24.70	32
3.533	25.19	36
3.325	26.79	21
3.230	27.59	7
3.151	28.30	26
3.121	28.58	20
3.085	28.92	9
2.944	30.33	11
2.901	30.79	7
2.803	31.90	6
2.729	32.80	8
2.692	33.26	11
2.656	33.72	9
2.594	34.55	10
2.554	35.11	13
2.476	36.25	14
2.433	36.92	14
2.407	37.32	17
2.321	38.77	11
2.279	39.51	9

POWDER PATTERN (DIFFRACTOGRAM) Figure 4

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid -hydrogensulfate

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POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid mesylate

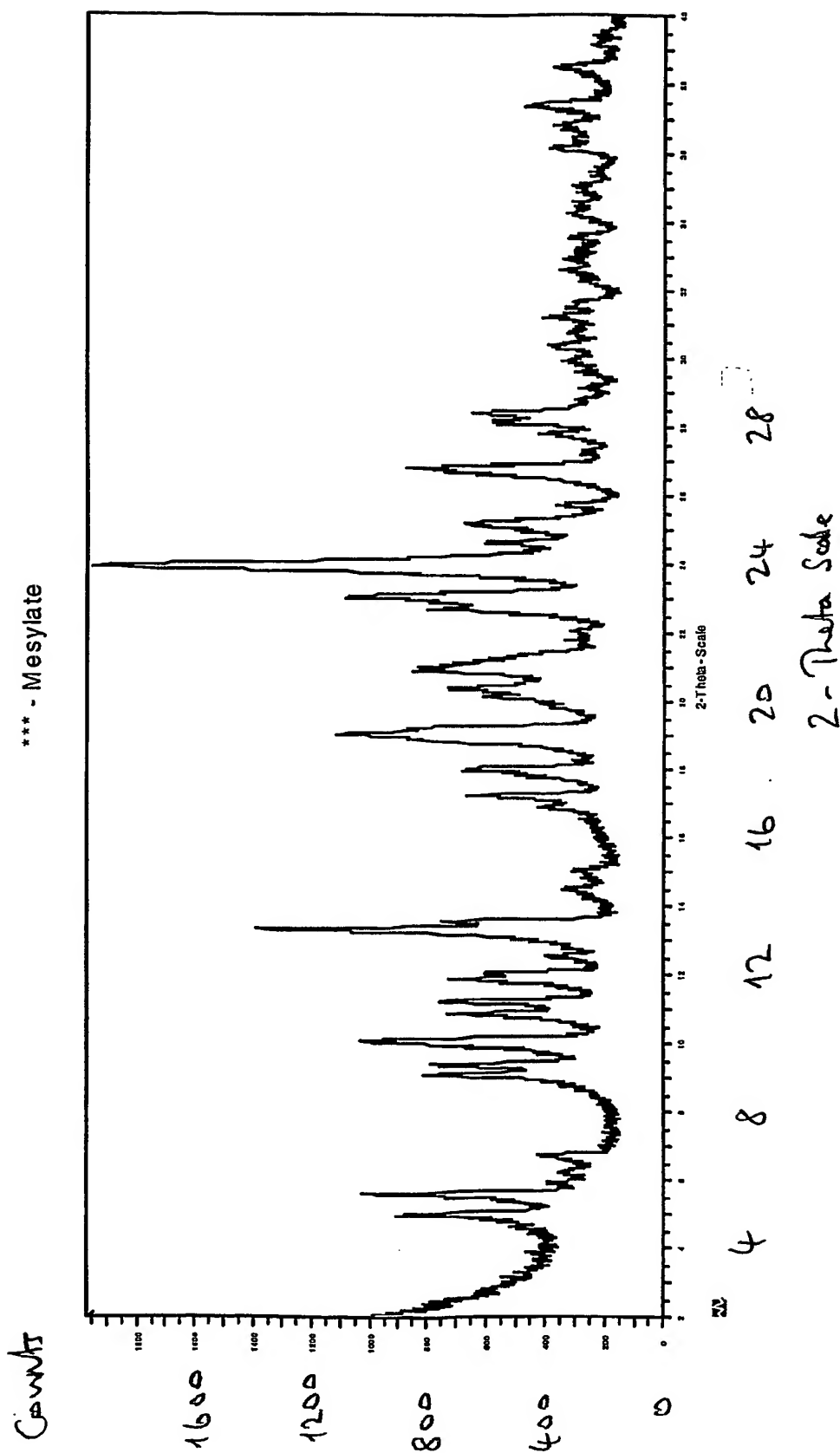
Table 5

MESYLATE		
d value (Angstrom)	Angle (° Cu Kα)	Intensity (Rel. Int.)
17.809	4.98	32
15.689	5.84	42
15.013	5.88	7
14.267	6.19	7
13.159	6.71	12
9.728	9.09	38
9.408	9.39	35
8.804	10.04	47
8.155	10.84	31
7.868	11.24	33
7.458	11.88	31
7.344	12.04	24
7.074	12.60	13
6.650	13.30	69
6.515	13.58	32
6.102	14.51	10
5.878	15.08	8
5.259	16.85	14
5.156	17.18	28
4.879	17.80	21
4.837	17.95	28
4.667	19.00	53
4.615	19.22	38
4.459	19.89	13
4.405	20.14	23
4.359	20.38	32
4.233	20.97	37
4.180	21.34	20
4.022	22.08	9
3.920	22.67	36
3.853	23.07	50
3.716	23.89	100
3.615	24.81	24
3.558	25.01	20
3.528	25.22	28
3.482	25.71	11
3.325	26.79	37
3.288	27.29	7
3.208	27.81	15
3.161	28.21	23
3.134	28.48	27
3.053	29.23	5
3.010	29.68	7
2.983	29.93	10
2.938	30.40	13
2.904	30.77	9
2.882	31.23	13
2.829	31.60	9
2.768	32.34	7
2.744	32.61	10
2.718	32.85	10
2.683	33.63	8
2.619	34.21	8
2.589	34.61	8
2.554	35.11	8
2.524	35.54	5
2.478	36.22	13
2.464	36.43	9
2.438	36.83	12
2.403	37.39	17
2.379	37.78	4
2.337	38.50	11
2.301	39.11	4
2.281	39.48	3

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POWDER PATTERN (DIFFRACTOGRAM) Figure 5

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid -mesylate



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POWDER PATTERN (D-I-LIST)
7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid sulfate

Table 6

SULFATE		
d value (Angstrom)	Angle (° Cu Kα)	Intensity (Rel. Int.)
17.924	4.93	15
13.099	6.74	20
9.795	9.02	12
8.806	10.04	48
7.346	12.04	63
6.667	13.27	72
6.546	13.52	51
6.103	14.50	21
5.953	14.87	19
5.377	16.47	6
4.888	18.13	39
4.661	19.03	46
4.467	19.86	19
4.328	20.51	21
4.203	21.12	32
3.859	23.03	44
3.698	24.05	100
3.616	24.60	32
3.542	25.12	35
3.467	25.68	13
3.333	26.72	21
3.246	27.46	11
3.158	28.23	33
3.125	28.54	21
3.070	29.06	12
2.954	30.23	10
2.907	30.73	7
2.844	31.43	3
2.814	31.77	5
2.722	32.88	8
2.695	33.22	10
2.659	33.69	7
2.603	34.42	9
2.561	35.01	11
2.483	36.15	10
2.435	36.89	12
2.408	37.32	14
2.327	38.67	9
2.288	39.35	5

POWDER PATTERN (DIFFRACTOGRAM) Figure 6

7-[2-(2-Aminothiazol-4-yl)-2-(methylcarbonyloxyimino)acetamido]-
3-vinyl-3-cephem-4-carboxylic acid -sulfate

